Effect of Silanization on Push-out Bond Strength of H₂O₂-etched Fiber Posts Using Various Resin Cements: An *In Vitro* Study

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ABSTRACT

Background and objectives: Etching of a fiber post is necessary for enhancing its adhesion to both the core and the root canal dentin. Many surface treatments are recommended for this purpose. The purpose of the present study was to evaluate the efficacy of silanization on the push-out bond strength of H₂O₂-etched fiber posts to a root canal dentin using different luting agents.

Materials and methods: Sixty maxillary central incisors and fiber posts were taken and grouped, based on surface treatment posts received, as group I (control); group II and group III—etched with 24% H₂O₂, and sub-grouped (IIa Ib; IIIa IIb; IIIa IIIb) based on the luting agent used. Group III received an additional treatment with silane before cementation and after etching. Each tooth was sectioned into three slices of 2 mm representing coronal, middle, and apical section of the tooth and subjected to a push-out test and the values obtained are statistically analyzed using two-way ANOVA and Tukey's *post hoc* tests.

Results: Among all the groups, group III irrespective of sub-groups showed the highest bond strength values followed by group II and then group I. Coronal region showed higher values than middle and apical.

Conclusion: Both the H₂O₂ etched groups (II and III) showed higher values than control group (group I). Silane treated groups (IIIa, IIIb) showed higher values than non-silanated group (IIa, IIb). There was no significant difference between the sub-groups (a, b) in all individual groups. In all groups, bond strengths were higher in coronal root region followed by middle and then apical.

Keywords: Fiber post, Hydrogen peroxide etching, Push-out bond strength, Scanning electron microscope.

Journal of Operative Dentistry and Endodontics (2019); 10.5005/jp-journals-10047-0071

INTRODUCTION

Trauma to the oral cavity can lead to fracture of teeth. Uncomplicated and complicated crown fracture is the most common traumatic dental injury to permanent teeth, and the teeth most commonly affected by trauma are the maxillary incisors, with a reported share of 96% of all the crown fractures (80% central incisors and 16% lateral incisors).¹ This is attributable to their anterior position and protrusion caused by the eruptive pattern.² When a greater amount of crown structure is lost, endodontic treatment followed by restoring the esthetics and function by a suitable coronal restoration is an important goal for a successful treatment.

Fiber posts and resin-based materials can behave as a mechanically homogeneous complex with dentin; their clinical use introduced a new restorative concept, ensuring high resistance to occlusal loading. This assumption has been recently supported by several *in vivo* and *in vitro* investigations.^{3–5,7–9}

Formation of a monoblock is essential for the success of a post luted to the root canal dentin. Improper bonding at the dentin/cement or the cement/post interface leads to an increased stress to occlusal loading, leading to the failure of post-retained restorations. A bonding between the resin cement and the fiber post is done using micromechanical and chemical means.^{11–14} The fiber post is covered by an epoxy resin that is highly cross-linked and has a high degree of conversion.¹⁵ Therefore, roughening the post to improve the micromechanical bonding has been recommended.¹²

Many techniques such as sandblasting and etching with hydrofluoric acid were used to improve bonding,^{13,16,17} but these methods caused damage to the structure of glass fibers and affected the integrity of posts. Agents that dissolve only the epoxy matrix

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How to cite this article: Majeti C, Veeramachineni C, *et al.* Effect of Silanization on Push-out Bond Strength of H₂O₂-etched Fiber Posts Using Various Resin Cements: An *In Vitro* Study. *J Oper Dent Endod* 2019;4(1):27–32.

Source of support: Nil

Conflict of interest: None

portion without interfering with fiber integrity were studied;^{14,18–20} potassium permanganate, sodium ethoxide, and hydrogen peroxide were used to etch and expose the glass fibers.^{12,14,18–20}

Chemical surface treatment protocols using bifunctional silanes have been tried to improve the bond strength¹¹ between the post and luting agent, but it revealed contradictory results.^{11,17,21–23}

Thus the aim of the study was to evaluate the efficacy of silanization on the push out bond strength of 24% H₂O₂-etched fiber posts to the root canal dentine using various luting agents.

The null hypothesis tested was

- Silanization does not influence the bond strength of the resin cement to the fiber post.
- Two different luting agents do not influence the bond strength of the post to root canal dentin.

- Root region does not have any influence on the bonding of the post to root dentin.

MATERIALS AND METHODS

Tooth Selection and Preparation

In the present study, 60 freshly extracted human maxillary central incisors free of caries, cracks, fractures, resorption, with fully matured roots and without previous root canal treatments or posts were selected. Teeth with similar sizes were selected and stored in 0.2% thymol solution until preparation. The crowns were de-coronated below the cemento-enamel junction, with a diamond saw under water coolant such that all the roots were standardized to a length of 12 mm using an electronic digital caliper (Radioshock).

Root Canal Treatment

The cleaning and shaping procedure was done using a ProTaper file system (Dentsply, Mallifler, Ballaigues, Switzerland) up to #F3 file and obturation was done with gutta-percha points (Maillefer Dentsply, Ballaigues, Switzerland) by the lateral condensation method using an AH-Plus sealer (Dentsply, Germany). A provisional restorative material (Cavit) was used for filling the coronal root canal orifices, and then to ensure the setting of the used sealer, the teeth were stored for one week in 100% humidity at 37°C.

Post Space Preparation

After one week, the gutta-percha in the coronal aspect of each root was removed with a Gates Glidden drill #3 (Dentsply/Maillefer, Switzerland) so that, 4 mm gutta-percha was preserved in the apex to maintain the apical seal. Post spaces were prepared to a depth of 8 mm, from top of the sectioned root surface. To standardize the final shape of the post space, a no. 2 drill of this system was used as the last drill. Finally, the canals were irrigated with distilled water and dried with paper points.

All the canals were irrigated with 17% EDTA to remove the smear layer followed by final flush with distilled water and air dried. Totally, 60 fiber posts (RelyX™ fiber posts, 3M ESPE AG, Germany) of size-2 were taken for 60 teeth and are grouped according to the surface treatment they received and the type of luting agent used (Flowchart 1).

The luting agents used were RelyX Unicem-2 (3M ESPE AG, Germany) and G-Cem Auto mix (GC America Inc.), which are self-adhesive dual-cure luting agents available as 2 paste systems. The luting agents are carried into the canal using a lentulo spiral and a thin layer is coated onto the post's surface. Finally, all the posts are luted into the respective canals and excess was removed and were light cured from coronal to apical direction using an LED light (Blue phase C8, Ivoclar vivident) for 60 seconds in such a way that the tip of the light unit was in a close contact with the coronal end of the fiber post. All the samples were stored in sterile saline for 1 week until they are subjected to the push-out test.

Preparation of Root Samples for Push-out Test

Each root specimen was sectioned into 3 slices of 2 mm each with a diamond saw under the water coolant, so that the first slice represents the coronal, second slice represents the middle, and last slice represents the apical third of the root canal. The apical side of each specimen was marked and finally all the slices were subjected to the push-out test under a universal testing machine (Autograph AG 15, Shimadzu, Japan).

Each slice was mounted on a platform provided, which supports the dentin where the part with the post and the surrounding tooth surface is at the center. A plunger with a diameter of 1 mm is used to perform the push-out at a cross-head speed of 1 mm/second until the post debonded and the values are recorded in mega pascals automatically.

Bond strengths of all groups were compared by a two-way ANOVA and a pair-wise comparison was done by Tukey's multiple *post hoc* test at a significance level of $p < 0.005$.

Preparation of De-bonded Specimens for SEM

De-bonded specimens were examined under a scanning electron microscope (SEM) (Model No S-3700N, Serial No: 37071-14, Hitachi High Tech Science Systems Corporation) to see the type of failure mode whether it is adhesive (between post and cement)/adhesive (between cement and dentin) or cohesive (within the cement).

For this purpose, each specimen was immersed in 96% ethanol for 2 minutes to dehydrate the specimen and then air dried. Slices were then mounted on aluminum stubs for gold sputtering (JOEL-JCF-110E) and observed under SEM at a magnification of 50x and 300x to evaluate the mode of failure.

RESULTS

Comparison of the Control Group with Experimental Groups

The push out bond strength values of group II and III (experimental groups) were higher than group I (control group) ($p < 0.005$), which was statistically significant, indicating a positive effect on the surface treatment of fiber post with 24% H₂O₂.

Comparison of Subgroups (a, b) (Luting Agents)

The pairwise comparison of subgroups (a, b) according to Tukey's multiple *post hoc* analysis showed no significant difference in the push-out bond strength values between both the sub-groups ($p > 0.005$) in all the groups (I, II, III) and at all root regions, predicting that the type of luting agent do not alter the bonding efficacy of the fiber post to the root canal dentin.

Comparison of Groups in the Coronal Root Region

The push-out bond strength values of all the groups at coronal level were evaluated. The results showed that group IIIa and IIIb had the highest bond strength values, which were statistically

Flowchart 1: Grouping: based on surface treatments and luting agents used

significant ($p < 0.005$) when compared with group Ia and Ib (control group). Though the bond strength values were high, they were not statistically significant when compared with group IIa and IIb (Fig. 1).

Comparison of Groups in the Middle Root Region

The push-out bond strength values of all the groups at the mid-root level were evaluated. The results obtained showed that group IIIa and IIIb had the highest bond strength values, which were statistically significant ($p < 0.005$) when compared with the other two groups (group Ia, Ib; group IIa, IIb), indicating the efficacy of etching and silanization in improving the adhesion (Fig. 2).

Comparison of Groups in the Apical Root Region

The push-out bond strength values of all the groups at the apical root level were evaluated. The results obtained were group IIIa and IIIb had the highest bond strength values, which were statistically significant ($p < 0.005$) when compared with the other two groups (group Ia, Ib; group IIa, IIb), indicating the efficacy of etching and silanization in improving the adhesion (Fig. 3).

Comparison of Bond Strength at Three Root Regions

The push out bond strength values evaluated statistically according to the root region (i.e., coronal, middle, and apical) by a two-way ANOVA revealed that there was a significant difference ($p < 0.005$) between the push out bond strength values of the three regions irrespective of the groups (I, II, III) and sub-groups (a, b). This indicates that the bond strength varies according to the root region irrespective of the surface treatment or the luting agent used.

Failure Mode Analysis

Adhesive failures between post and cement were less in group IIIa and IIIb when compared with all other groups, indicating an improvement in adhesion between the post to the cement in group III (Fig. 4).

Cohesive failures are more in group IIIa and IIIb when compared with other groups, indicating an increased effect of bonding capability of luting agents after etching and silane application.

Fig. 1: A comparison of three groups I, II, III (control, without silanization and with silanization) and two subgroups a, b (RelyX and G-Cem) in the coronal root region

Fig. 2: A comparison of three groups I, II, III (control, without silanization and with silanization) and two subgroups a, b (RelyX and G-Cem) in the middle root region

Fig. 3: Comparison of three groups I, II, III (control, without silanization and with silanization) and two subgroups a, b (RelyX and G-Cem) in the apical root region

Figs 4A to C: Scanning electron microscopic images of types of failure: adhesive failure between dentin and cement, adhesive failure between cement and post, cohesive failure within the cement. (A) Scanning electron microscopic images of types of failure; (B) Adhesive failure between dentin and cement; (C) Adhesive failure between dentin and cement

In all groups (I, II, III), irrespective of the type of surface treatment or luting agent, more number of adhesive failures between dentin and luting agent were noted, showing that it was the weakest link.

DISCUSSION

Adhesive posts rely on the strength of bond between different interfaces for their retention. Among them, the interface between root dentin and resin cement has been the object of several studies involving bond strengths^{25–27,30,31} and microscopic investigations.^{28,29} Since the introduction of prefabricated FRC posts, a continuous effort has been made to improve the bonding potential of adhesive systems to root canal dentin, as radicular dentin has been shown to offer far less favorable conditions for adhesion than coronal dentin and enamel.³⁰

Maxillary central incisors are chosen in the present study since they are the teeth most commonly prone to trauma owing to their position in the dental arch.¹

Results of the present study showed a significant improvement of bond strength values in both the experimental groups when compared with control groups, showing the efficacy of etching the fiber posts with 24% H₂O₂ for 10 minutes. These results are in accordance with the study of Monticelli et al.¹⁰

Fiber posts are covered by epoxy resin, which has a high degree of conversion and few reactive sites to chemically bond to the resin cement.¹⁵ So, to improve the bond between post and adhesive resin, removal of epoxy layer is essential without disturbing the fiber integrity. Unlike the use of corrosive forms of industrial epoxy resin etching techniques such as sodium ethoxide or potassium permanganate, 24% H₂O₂ etching proved to be effective by causing no damage to the fibers by removing only the superficial epoxy layer. Dissolution of epoxy resin probably rely on an electrophilic attack of the H₂O₂ to the cured secondary amine. Thus the spaces created between the fibers provide conditions for the micro-mechanical interlocking of the resin with the post.³⁰ Furthermore, the exposed fibers become available to chemically bond to adhesive through silane agent. This is the reason for attaining higher values in experimental groups that are subjected to H₂O₂ etching than in control groups.

The efficacy of silanization in improving the adhesion of fiber post is a contradictory issue since few authors support it^{11,19} and others oppose it^{25,26} saying it is clinically negligible. Therefore, the present study aimed at evaluating the role of silanization in post-adherence to radicular dentin. Among all the groups, the silanated groups showed higher bond strength values than in all the root regions irrespective of the luting agent used, showing the effectiveness of silanization in improving the adhesion, thus rejecting the first null hypothesis. These results are in accordance with previous studies showing the positive role of silane in enhancing the bond strength of a fiber post.^{11,19}

The improved interfacial strength achieved following silanization may depend on the reaction between silane molecules and the silica component in the quartz fibers.³⁰ In this study, the silane coupling agent used was 3-methacryloxypropyltrimethoxysilane; the methacryloxy group in it probably enables the chemical union between methacrylate groups in the exposed fibers as well as methacrylate groups of the luting agent.

The chemical bonding according to chemical bonding theory is by formation of covalent bonds from the reaction of

organo-functional group (R) and the hydrolyzed alkoxy groups (R'O)₃ respectively, with resin matrix and mineral substrate of FRC post. Moreover, once an intimate contact occurs between the interfacing materials, the van der Waals' forces also become effective along with chemical bonds, improving the bonding effectiveness.¹¹ That is the probable reason why we got higher values in silanated groups when compared to the remaining groups.

Our results show that there is no significant difference between both the luting agents in all the groups irrespective of the root regions, thus accepting the second null hypothesis and help us to infer that the type of self-adhesive luting agent does not influence the adhesion.

RelyX Unicem-2 is a methacrylate based self adhesive resin cement containing phosphate groups (i.e.) acidic monomer that demineralizes the teeth and create micromechanical retention and chemical adhesion to hydroxyapatite. G-Cem is methacrylate and fluoroaluminosilicate-glass-based self-adhesive resin cement containing a phosphoric ester monomer and was claimed to have higher bonding abilities than other self-adhesive cements. But the results showed that in all the groups, irrespective of the root region, there is no significant differences between both luting agents. The reason may be that both luting agents etch and bond to the dentin by a micromechanical means owing to the action of phosphate groups, and upon comparing them individually, they showed greater values in silanated groups, proving the formation of a chemical bond between the silane agent and luting agent. The results indicate a probable chemical interaction between adhesive cement and hydroxyapatite of root canal as explained by Bitter et al., who showed the ability of self-adhesive cements to hybridize the root dentine.²²

The push out test was preformed since it produces shear stress at the interface between the post and cement, which mimics the stresses under a clinical situation.¹⁷

Results of the present study show that there is significant difference in the bond strength values among the coronal, middle, and apical root regions in all the groups irrespective of the surface treatment and the luting agent used, thus rejecting the third null hypothesis.

These results are in accordance with the previous studies that proved that the region of root canal significantly affects bond strength.^{19,21,25,30} The reason for this might be the decrease in the tubule density from the coronal to the apical region, as explained previously by Ferrari et al.³⁰ But the most likely explanation for this higher resistance to dislodgement of post in coronal region and decreased resistance in apical region could be due to decreased effectiveness of the curing light to penetrate from coronal region to the apex.²⁸ Also the reduction in the bond strength may be related to the difficulties of moisture control in apical third of post space, which may cause incomplete infiltration of the resin cement. The reduction in bond strength in middle and apical region might also be related to unequal distribution of resin cement and void formation within the cement^{18,21} or due to traces of gutta-percha and endodontic sealer that may remain in these thirds after postspace preparation.²²

The analysis of failure modes under SEM revealed that predominant failures are adhesive failures between the luting agent and dentin irrespective of the group (control or experimental), which is in accordance with a number of *in vivo* and *in vitro* studies^{6,23,30} showing that it is the weakest link.

The H₂O₂ etched silanated group irrespective of the type of luting agent showed least adhesive failures between the post and luting agent compared with control groups and H₂O₂-etched non-silanated groups, signifying the important role of etching and silanization in improving bond strength, which is in accordance with the previous studies supporting the efficacy of silane agent application to the fiber post.^{19,24}

Also, a greater number of cohesive failures are seen in all groups and subgroups, indicating that self-adhesive cements bond effectively to both the dentin and the post, which is in accordance with the study by Sahmal et al., who stated that higher bond strengths result in a higher percentage of cohesive failures.²⁰

CONCLUSION

Within the limitations of the present study, it can be concluded that:

- Hydrogen peroxide (H₂O₂) etching of fiber post would be the recommended method for effective bonding of the post to resin cements.
- Silanization had a major role in improving the adhesion of the fiber post to resin cement and to the root dentin after H₂O₂ etching.
- The two types of cements do not have any influence in altering the bond strength of the fiber post to root canal dentin.
- Root region significantly affects the bond strength of the fiber post to root dentin.

Further *in vivo* and *in vitro* studies are necessary to support the efficacy of silanization; moreover, other strategies to improve the bonding of the fiber post to luting agent and to the root dentin should be investigated.

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